

XV. *Experiments to shew the Nature of Aurum Mosaicum: By Mr. Peter Woulfe, F. R. S.*

Read, Feb. 14. 1771. **T**HE Aurum mosaicum is known by the names of Aurum musivum, Aurum musicum, and Purpurina. We can collect but very imperfect ideas of this preparation, from the writings of antient and modern chemists. It is true, that the method of making it has been described by many; but no one hitherto has led us into the knowledge of its nature, by a sufficient number of experiments. It has been much used formerly as a pigment, but is now almost laid aside, and the Bronzes substituted in its place. It is sometimes used in medicine as a vermifuge, but how improper and uncertain it is for that purpose, will appear by the following experiments.

The best preparation described hitherto for making Aurum mosaicum, is set down in the London Dispensatory, and is as follows.

Take of tin twelve ounces, of flowers of sulphur seven ounces, of sal ammoniac six ounces, and of purified mercury six ounces; melt the tin, and add the mercury to it; and when cold, powder it, and mix with it the sal ammoniac and sulphur. Sublime the mixture in a matrafs. The Aurum mosaicum will

will be found under the sublimate, with some drops at the bottom \*.

*Ætiology of the Operation.*

As soon as the mixture grows warm, the tin acts on the sal ammoniac, and sets free its volatile alkali; and this, having a great affinity with sulphur, joins with a great part of it †, rises in the sublimation, and is totally dissipated. The portion of tin, which acted on the Sal ammoniac and set free its volatile alkali, unites with the acid of salt of the sal ammoniac, and forms a salt of tin, which sublimes ‡. The mercury, which was added only in order to divide the tin, unites with some of the sulphur, and likewise sublimes and forms a cinnabar. The tin which remains, unites with the remaining sulphur, and forms the Aurum mosaicum, which is found in the bottom of the matrafs. Instead of performing this operation in a matrafs, I have used a glass retort, fixed in a black lead crucible, with sand round it; the crucible was put into a proper furnace, and a charcoal

\* The proportion formerly used was equal parts of each, which in the quantity of tin here employed, viz.  $\text{℥ } 12$ , produced only  $\text{℥ } 13\frac{1}{2}$  of Aurum mosaicum; whereas the same quantity of tin in the proportion of the London dispensatory afforded  $\text{℥ } 16$ .

Troy weight was made use of in all the following experiments.

† Sulphur combined with volatile alkali forms a volatile liver of sulphur, called by Mr. Boyle volatile tincture of sulphur and quick lime.

‡ Filings of tin, calx of tin, or tin divided by amalgamation with mercury, distilled with sal ammoniac, &c. decomposes it, whereby its acid of salt unites with the tin, and its volatile alkali is set free.

fire made round it, an adopter was luted to the retort, and to the adopter a quilled receiver; a long vial was fitted to the quill of the receiver, in order to hold the liquor that distilled.

It will be easy by this apparatus to perform the operation without any considerable loss, provided the fire be well regulated. It is necessary to make a very slow fire at first, in order to condense the volatile fumes; for a great quantity of air is set free, when the ingredients begin to act on one another. The fire should be rather slow for the first four or five hours, and then gradually increased, until the crucible becomes moderately red hot; in which state it is to be continued during the rest of the operation, which commonly lasts about sixteen hours from the beginning to the end.

If this operation be performed for the sake of the Aurum mosaicum only, or for its sublimate, it may as well be done in a matrafs or body; and in that case there is no great loss of the sublimate.

Twelve ounces of tin with sulphur, sal ammoniac and mercury in the proportion of the London dispensatory, prepared in this manner produced,

	3 3 3
* Of volatile liver of sulphur, liquid and dry	1 4 2
Of sublimate found in the retort and adopter	13 2 0
Of Aurum mosaicum	16 0 0
Loss in the operation	0 1 1
Weight of all the ingredients	33 1 0 0

\* The volatile liver, for the most part, comes over liquid, and is often found in the long vial in form of most beautiful ramifications, which are a crystallization of the volatile liver.

The

This operation was often repeated, and always with some little variation in the products, owing to the management of the fire, which cannot be always made alike.

If this preparation be made with too great a degree of fire, the Aurum mosaicum will be partly melted, or of a dark colour ; and if the fire be not continued for a sufficient time, a portion of the cinnabar and salt of tin will remain with the Aurum mosaicum.

*The Sublimate of Aurum mosaicum examined.*

The sublimate, which was obtained in preparing the Aurum mosaicum weighing  $\frac{3}{4}$  13  $\frac{3}{4}$  2 was finely powdered and digested with distilled water sharpened with some acid of salt \*, and when cold was filtered ; more water was added to what remained in the filter, by which means it was deprived of its soluble part.

† The undissolved part of the sublimate dried, and then sublimed in a retort, produced  $\frac{3}{4}$  9  $\frac{1}{2}$  of cinnabar, which was of a dark colour, owing to an excess of sulphur ; there came over into the receiver a small quantity of an acid liquor, and there was found in the retort  $\frac{3}{4}$   $\frac{1}{2}$  of Aurum mosaicum, which added to the former quantity makes  $\frac{3}{4}$  16  $\frac{1}{2}$ .

The soluble part of the sublimate is composed of tin united to the acid of salt ; in order to know the quantity of tin which it contains, a sufficient quantity of fixed alkali dissolved in water was added

\* Salt of tin mixed with water becomes turbid, and a portion of the tin precipitates ; therefore the acid of salt was added, to prevent the precipitation.

† This contains some running mercury ; but in the sublimation it disappears, and forms cinnabar.

to it, by which means the tin was precipitated; this precipitate weighed  $\frac{3}{2} \frac{3}{7}$ .

An ounce of tin dissolved in the acid of salt \*, being precipitated with a solution of fixed alkali in water, well washed and dried † weighed  $\frac{3}{2} \frac{1}{4}$ ; so that a precipitate of tin contains only  $\frac{4}{5}$  of tin, and therefore the  $\frac{3}{2} \frac{3}{7}$  of precipitate obtained from the sublimate contain only  $\frac{3}{2} \frac{3}{2} \frac{1}{2}$  nearly of tin; this being deducted from  $\frac{3}{2} \frac{1}{2}$ , the quantity of tin used in the operation, makes  $\frac{3}{2} \frac{9}{5} \frac{1}{2}$ , which is the real quantity of tin contained in the  $\frac{3}{2} \frac{1}{6} \frac{1}{2}$  of Aurum mosaicum obtained in this process; therefore  $\frac{3}{2} \frac{1}{10}$  of Aurum mosaicum contains  $\frac{3}{2} \frac{1}{2}$  of tin, and  $\frac{3}{2} \frac{7}{10}$  of sulphur; for  $\frac{3}{2} \frac{9}{5} \frac{1}{2}$  of tin is to  $\frac{3}{2} \frac{1}{6} \frac{1}{2}$  of Aurum mosaicum, as  $\frac{3}{2} \frac{1}{2}$  of tin is to  $\frac{3}{2} \frac{1}{10}$  nearly of Aurum mosaicum. This will be further illustrated by other experiments.

The tin, which was precipitated by adding a fixed alkali to the soluble part of the sublimate, was distilled with iron filings and fixed alkali; but no mercury was obtained. This shews that none of the mercury unites with the acid of the sal ammoniac.

There was no volatile alkaline smell produced by the addition of fixed alkali to the soluble part of the sublimate, though there were an excess of it added; which proves that the sal ammoniac was totally decomposed.

\* The vapour, which arises in dissolving tin in the acid of salt, becomes inflammable, when the solution is made in large quantity, by means of heat; the like happens also with regard to lead.

† This precipitate, if dried with too much heat, takes fire, and burns like a dried plant that contains nitre.

The

The soluble part of the sublimate of Aurum mosaicum produces crystals of an irregular form, which do not deliquesce in the air like all other salts of tin, owing chiefly to their having a less portion of acid. A drop of the solution of this sublimate, crystallised on a piece of glass, and viewed with a microscope, has very much the appearance of the crystals of alum.

Aurum mosaicum, when well prepared, is of a shining golden colour, has no taste, and is not soluble in water. It is not acted on by acids, nor by fixed or volatile alkalis dissolved in water. If melted with an equal quantity of fixed alkaline salt of tartar, it forms a liver of the colour of gumbouge, which is, for the most part, soluble in water, and may be precipitated by any acid.

If Aurum mosaicum be distilled with iron filings, no mercury will be obtained.

It is well known, that tin deflagrates violently with nitre; therefore it will not seem surprising, that the Aurum mosaicum should have that property in a far greater degree, it being a composition of tin and sulphur\*.

Sulphur, combined with metallick substances, renders them inactive, as we see in cinnabar, antimony, &c.; therefore Aurum mosaicum well prepared must be a very unfit medicine for worms.

Aurum mosaicum is often found to have a very rough taste; but that is owing to the salt of tin, which has not been sufficiently dissipated in the sublimation, and, in that state it may effectually destroy

\* May not this substance be useful in fire-works, as also sulphurated iron?

worms; but it must be allowed to be a very uncertain medicine, and perhaps dangerous, as it may contain too small or too great a quantity of the salt of tin.

If salt of tin be good for worms, it would be right to ascertain its dose, and give it in a proper vehicle.

*Tin combined with Sulphur, by Fusion.*

Four ounces of tin melted and saturated with sulphur weighed 35, and formed a black shining flaky brittle substance when melted. The tin need not be made quite red hot for this operation; for, when the sulphur is mixed with it, a deflagration ensues, and the mixture grows red hot. In order to saturate the tin with as much sulphur as possible, it must be added to it at two or three different times. The tin, notwithstanding, cannot be, by this means, perfectly saturated with sulphur; for after powdering and sifting it, there remains in the sieve a portion of the tin, which will flat under the pestle, and not powder any more, unless melted and combined with more sulphur. If this operation be done with too great a degree of fire, the increase of weight will not be so considerable, on account of the great fire, which dissipates some of the sulphur.

Four ounces of Aurum mosaicum melted in a covered crucible loses 36 of its weight, and becomes a mass somewhat like melted sulphurated tin, though it be not so shining, nor so flaky, but rather more of a needle form. If the fusion be repeated two or three times, some of its sulphur will be each time dissipated, and have exactly the appearance of sulphurated tin.

**Aurum**

Aurum mosaicum melts much more readily than sulphurated tin; and that, because it contains a greater quantity of sulphur.

Sulphurated tin, by calcination, is totally deprived of its sulphur and phlogiston.  $\frac{3}{4}$  of tin saturated with sulphur, and then carefully calcined weighed  $\frac{3}{4}\frac{1}{2}$ , so that a calx of tin prepared in this manner, weighs  $\frac{1}{5}$  more than the tin it contains\*.

Four ounces of Aurum mosaicum calcined in the same manner, weighed  $\frac{3}{4}$  3,  $\frac{3}{4}$  2,  $\ominus$  1, which, being a calx of tin, of the nature of that made with sulphurated tin, contains  $\frac{1}{5}$  less of tin, which  $\frac{1}{5}$  being deducted, makes  $\frac{3}{4}$  2,  $\frac{3}{4}$  7,  $\ominus$  1, gr.  $4\frac{1}{2}$ , the quantity of tin contained in  $\frac{3}{4}$  4 of Aurum mosaicum†. An ounce of Aurum mosaicum carefully calcined, and reduced with flux, produced only  $\frac{3}{4}$   $\frac{1}{2}$  and gr. 11 of tin: we may conclude from the foregoing experiments, that  $\frac{3}{4}$  1 of Aurum mosaicum contains more than this quantity of tin; but it is well known that metallick bodies, which have been much calcined, and especially tin and zinc, always lose in their reduction.

Aurum mosaicum will be of a black colour, if too small a quantity of sulphur be used, and if the fire be too strong and too long continued†.

\* All metallick substances, and even zinc, though a good deal of it dissipates in flowers, increase in weight by calcination.

† It is almost impossible to calcine Aurum mosaicum, without some loss; for it is so light and subtil, that it cannot be stirred without dissipating some of it; therefore the quantity of tin which it contains, cannot be exactly ascertained by this means.



*Receipts for making Aurum Mosaicum without Mercury.*

1st. Take  $\frac{3}{4}$  8 of granulated tin sifted through a fine sieve, and mix it well with  $\frac{3}{4}$  6 of sulphur, and  $\frac{3}{4}$  4 of sal ammoniac; put the mixture into a matras or body, and calcine it for six or seven hours. The Aurum mosaicum hereby obtained is not of so bright a colour, as that made in the usual manner; and that, on account of the tin not being sufficiently divided in order to unite well with the sulphur.

2d. Take  $\frac{3}{4}$  8 of tin reduced to a calx by calcination, and mix it well with  $\frac{3}{4}$  7 of sulphur, and  $\frac{3}{4}$  4 of sal ammoniac; calcine it as the former. This makes a good coloured Aurum mosaicum, though it be found here and there of an unequal colour, owing to some of the tin, which had been too much calcined, and thereby prevented from uniting with the sulphur.

3d. Take  $\frac{3}{4}$  8 of tin, and saturate it by fusion with sulphur, powder and mix it well with  $\frac{3}{4}$  5 of sulphur, and  $\frac{3}{4}$  4 of sal ammoniac; calcine the mixture as before. This produces a good coloured Aurum mosaicum.

*Receipts for preparing Aurum Mosaicum, without Mercury or Sal ammoniac.*

1st. Take  $\frac{3}{4}$  8 of tin, saturate it by fusion with sulphur, and mix it with  $\frac{3}{4}$  4 more of sulphur, and calcine as before, but with a less degree of fire. This forms rather a dark coloured Aurum mosaicum, owing to the sulphur which melts, and in great  
measur e

measure separates and swims on the surface of the sulphurated tin.

2d. Take  $\frac{3}{4}$  10 of sulphurated tin, powder and mix it well with  $\frac{3}{4}$  4 of sulphur, and  $\frac{3}{4}$  2 of spirit of salt; calcine the mixture as the former. This forms an Aurum mosaicum of a tolerable good colour. This mixture soon grows warm of it self after being put into the body, and produces a penetrating vinous smell, no ways like that which is afforded by dissolving tin in the acid of salt.

In this operation, part of the tin unites with the acid of salt, and is at last dissipated in the operation.

3d. Take  $\frac{3}{4}$  8 of tin, and saturate it with sulphur, powder and mix it with  $\frac{3}{4}$  5 of sulphur; put it into a body, and pour on it  $\frac{3}{4}$  2 of volatile liver of sulphur obtained in making Aurum mosaicum, and calcine it as fore. This does not make so good a coloured Aurum mosaicum as the former, owing to the fusion of the sulphur, which in great measure separates from the sulphurated tin. Soon after the volatile liver was poured on the mixture of sulphurated tin and sulphur, it grew so hot that the body could scarcely be held in the hand.

4th. Take  $\frac{3}{4}$  4 of tin, saturate it with sulphur, powder and mix it well with  $\frac{3}{4}$  2 of sulphur, and  $\frac{3}{4}$  1 of tin dissolved in the acid of salt and crystallised; calcine it as usual. This produced  $\frac{3}{4}$  6½ of very good and quite tasteless Aurum mosaicum, so that  $\frac{3}{4}$  4 of tin by its union with sulphur encreased in weight  $\frac{3}{4}$  2 ½.

This operation was done in a retort, to which there was luted an adopter and receiver, in order to collect

the salt of tin, which for the most part came over congealed.

5th. Take  $\frac{3}{4}$  10 of sulphurated tin, powder and mix it well with  $\frac{3}{4}$  16 of corrosive mercury sublimate; put it into a retort, to which an adopter and receiver is to be luted, calcine it for six hours, at first with a middling fire, and for the last three hours the retort must be red hot.

In this operation, a portion of the tin unites with the acid of the mercury sublimate; and forms the smoaking liquor of Libavius, which distills for the most part in a liquid form; the mercury contained in the mercury sublimate unites with a small portion of sulphur (about  $\frac{1}{7}$  of its weight), and sublimes in form of cinnabar in the top of the retort; the remaining tin, having a sufficiency of sulphur, forms the Aurum mosaicum, which is found at the bottom of the retort of a most beautiful sparkling golden colour.

The reason for obtaining Aurum mosaicum by this operation is, that the greatest part of the tin unites with the acid of the mercury sublimate, and rises in distillation; the remaining tin has thereby a sufficient quantity of sulphur to form the Aurum mosaicum.

Some of the foregoing receipts did not well succeed at the first or second trial; and many other experiments, which did not answer, have here been omitted.

From the foregoing experiments, we may certainly conclude, that Aurum mosaicum is a combination of tin and sulphur; it contains better than  $\frac{1}{3}$  of sulphur. It also appears that the only use of the  
mercury,

mercury is to divide the tin; and that the sal ammoniac serves only to prevent the fusion of the sulphur.

The following proportion will answer better than that of the London Dispensatory; for there will be a greater produce of Aurum mosaicum, though a less quantity of mercury and sal ammoniac be used.

Tin  $\frac{3}{4}$  12, sulphur  $\frac{3}{4}$  7, sal ammoniac  $\frac{3}{4}$  3, and mercury  $\frac{3}{4}$  3.

This proportion yields  $\frac{3}{4}$  17 $\frac{1}{2}$  of Aurum mosaicum, whereas that of the London Dispensatory gives only  $\frac{3}{4}$  16.

The soluble part of the sublimate of Aurum mosaicum answers far better for dying than any solution of tin; a small quantity of it with cochineal will dye silk, and especially cloth, of a fine scarlet colour; silk may be dyed of a fine crimson colour, by its means, with the addition of brazil wood, peach wood, or braziletto; but with logwood, silk and cloth may be made of a great variety of fine purple colours, which seem lasting.

The property, which this sublimate has of making finer colours than any solution of tin, engaged me to make many trials, with other preparations of tin; and I found, that when tin was united to the acid of salt, and distilled or sublimed, it would produce finer colours than any solution or combination of tin, un-sublimed or undistilled.

I must be excused for the present, for not telling the reason of this; it may be discovered by examining well the products, which are obtained by making the liquor fumans of Libavius, in the common manner.

May not iron and copper, united to the acid of salt and sublimed, answer better for dying, than other preparations of iron and copper?

Most other metallick substances may be after this manner more intimately combined with a greater portion of sulphur than by fusion. Bismuth is the only one, which produced a golden colour, and that not so fine a one as Aurum mosaicum. Iron, copper, lead, and regulus of antimony, produce black combinations; arsenic forms a reddish mass like realgar; zinc does not in this manner, nor in any other way that I know, combine with sulphur.

*An Apparatus for making Aurum Mosaicum in the cheapest manner.*

A glass vessel cannot be used for this operation more than once, because it is necessary to break it, to get out the Aurum mosaicum. The following utensil may be employed a great number of times, and save the expence of glass.

Take a black lead crucible, N°. 60; bore a round hole in its bottom about three inches diameter; and saw off an inch of its upper edge; if it has a lip, get a round piece of burnt clay, of an inch thick or rather more, to fit exactly into this edge; the composition, which is used for making paving-tiles, answers very well for this purpose. In order to make use of this apparatus, fit the round piece of burnt clay to the inner edge of the crucible, by means of some loam softened with glue, and dry it slowly; then turn it upside down, and lay it in a proper furnace on two iron bars. The mixture for the Aurum mosaicum is to be put in through the  
round

round hole at top, and then covered with an aludel and luted ; this serves to collect the flowers and the sublimate which rises. The fire is to be made under and all round the crucible. 11 lb. Troy of Aurum mosaicum may be made here at a time ; and when the operation is over, the bottom or round piece of burnt clay will easily come out with the Aurum mosaicum. A large crucible may be made use of, if a larger quantity be required to be made at once. The operation cannot fail of success, provided the fire be made of a sufficient strength, and of an equal degree from the bottom to the top of the crucible, which is easily done in a good furnace. The operation is finished in eight hours, unless the volatile liver is wanted.

White arsenic, digested with a solution of tin in the acid of salt, becomes soon black ; it does hereby regain its phlogiston, and is reduced to the state of regulus of arsenic, and will by this means readily combine with copper, and other metallick substances ; which it would not do, without the help of phlogistic substances. This is the most easy and ready way of reducing arsenic to its metallick form : the arsenic may be deprived of the solution of tin, which adheres to it by washing it with water. It is to be dried slowly, for otherwise it is apt to catch fire.

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*A Method of dying Wool and Silk, of a yellow colour, with Indigo ; and also with several other blue and red colouring Substances.*

THE Saxon blues have been known for some time ; and are made by dissolving indigo in oil of vitriol,

vitriol, by which means the indigo becomes of a much more lively colour, and is extended to such a degree, that it will go very far in dying.

A receipt for making the best Saxon blue will, I dare say, be agreeable to many; I will, therefore, give the following, which produces a very fine colour, and never fails of success.

Mix  $\frac{3}{4}$  1 of the best powdered indigo, with  $\frac{3}{4}$  4 of oil of vitriol in a glass body or matrafs: and digest it for one hour with the heat of boiling water, shaking the mixture at different times; then add  $\frac{3}{4}$  12 of water to it, and stir the whole well, and when grown cold filter it. This produces a very rich deep colour; if a paler blue be required, it may be obtained by the addition of more water. The heat of boiling water is sufficient for this operation, and can never spoil the colour; whereas a sand heat, which is commonly used for this purpose, is often found to damage the colour, from its uncertain heat.

Indigo, which has been digested with a large quantity of spirit of wine, and then dried, will produce a finer colour than the former, if treated in the same manner, with oil of vitriol.

No one, that I know of, has heretofore made use of the acid of nitre, instead of the acid of vitriol; and it is by means of the former that the yellow colour is obtained: it was nevertheless natural to use it, on account of its known property of making yellow spots, when dropped on any coloured cloth.

The acid of salt does not dissolve indigo, and therefore is of no use in dying.

*Receipt for making the yellow dye.*

Take  $\frac{3}{4}$  of powdered indigo, and mix it in a high glass vessel, with  $\frac{3}{4}$  2 of strong spirit of nitre, previously diluted with  $\frac{3}{4}$  8 of water; let the mixture stand for a week, and then digest it in a sand heat for an hour or more, and add  $\frac{3}{4}$  4 more of water to it; filter the solution, which will be of a fine yellow colour.

Strong spirit of nitre is liable to set fire to indigo; and it is on that account that it was diluted with water, as well as to hinder its frothing up.  $\frac{3}{4}$  2 $\frac{1}{2}$  of strong spirit of nitre will set fire to  $\frac{3}{4}$   $\frac{1}{2}$  of indigo; but, if it be highly concentrated, a less quantity will suffice.

If the indigo be digested twenty four hours after the spirit of nitre is poured on it, it will froth and boil over; but, after standing a week or less, it has not that property.

One part of the solution of indigo in the acid of nitre, mixed with four or five parts of water, will dye silk or cloth of the palest yellow colour, or of any shade to the deepest, and that by letting them boil more or less in the colour. The addition of alum is useful, as it makes the colour more lasting; according as the solution boils away, more water must be added.

None of the colour in the operation separates from the water, but what adheres to the silk or cloth; of consequence this colour goes far in dying.

Cochineal, Dutch litmus, orchel, cudbear, and many other colouring substances treated in this manner, will all dye silk and wool of a yellow colour.



The indigo which remains undissolved in making Saxon blue, and collected by filtration, if digested with spirit of nitre, dyes silk and wool of all shades of brown inclining to a yellow.

Cloth and silk may be dyed green with indigo; but they must first be boiled in the yellow dye, and then in the blue.